UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Marketing Service
Cotton Division

COTTONSEED

METHODS OF CHEMICAL ANALYSIS AND GRADE CALCULATIONS APPROVED BY THE DIRECTOR, COTTON DIVISION, AGRICULTURAL MARKETING SERVICE

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The regulations governing the inspection, sampling, and certification of cottonseed sold or offered for sale for crushing purposes (Code of Federal Regulations, Title 7, Part 61) and the official standards for grades of cottonseed (Code of Federal Regulations, Title 7, Part 28) require that the inspection, sampling, analyzing, and grading of cottonseed be performed as prescribed in methods approved from time to time by the Agricultural Marketing Service. This bulletin contains the approved methods for chemical analysis and grade calculations and supersedes the bulletin issued October 1952 on this subject.

June 28, 1954

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873514

METHODS FOR THE ANALYSIS OF COTTONSEED

Sec. 1: The Sample of Cottonseed.

(a) Laboratory Sample. The sample received at the laboratory shall consist of 2 pounds of cleaned seed. (See appendix for approved containers). It shall be sealed in an airtight container and shall be accompanied by a certificate by a licensed cottonseed sampler in the form approved by the Director, Cotton Div., Agricultural Marketing Service, U. S. Department of Agriculture.

(b) Sample for Moisture Test. Prior to handling the sample, the portion of seed for the original moisture test shall be withdrawn and immediately placed in a suitable container with a tight fitting cover.

(c) Handling of Sample. The remaining sample shall be examined by the chemist who shall correct the weights reported to him by the sampler, for such additional foreign matter as he may find in the sample by screening it and by picking out all other particles of foreign matter by hand. The sample shall then be placed in an approved mechanical mixer and handled according to directions for the use of the mixer used. The cleaned and mixed sample shall then be quartered, and one-half of it returned to the original container and retained as a referee sample for a period of time not to exceed two weeks. The second half of the sample shall be placed in an air-tight container and used by the chemist for his analysis. (See appendix for approved equipment)

Sec. 2: Original Moisture.

Determination. Weigh as rapidly as possible 2 samples of between 5 and 10 grams each from the portion reserved for moisture into tared moisture dishes, picking out and discarding all pieces of foreign matter. The uncovered dish containing the sample is placed in an approved type oven at 101°C. for from 12 to 16 hours, or most conveniently over night. The dish when removed from the oven is covered, cooled in an efficient desiccator and weighed, the

loss in weight being calculated as moisture. (See appendix for approved equipment).

Sec. 3: Fuming and Grinding.

(a) See appendix for approved equipment.

(b) Preparation of Seed for Oil and Ammonia Determination. Dry an approximately 60 gram portion, for 2 hours, in an approved type oven at 130°C. plus or minus 3°C.. Toward the end of this drying period, absorb into the inner walls and bottom of a porous earthenware pot, 1.5 ml. of concentrated hydorchloric acid. The acid is distributed all over the inside of the pot, and when absorbed, the inside of the pot must appear dry, otherwise a new pot must be substituted. (For fuming delinted cottonseed, use 1.0 ml. of concentrated hydorchloric acid.) Place the dried seed in the pot, cover and place in the fuming oven previously opened and ventilated for at least 5 to 10 minutes, and fume for 1 hour. The oven temperature should gradually rise to, but not exceed, 115°C.. The lint should be loose and brittle, not scorched. Grind the dried and fumed sample in an approved mill which has been adjusted to produce a fine meal. After grinding, open up the mill and carefully brush out all remaining ground seed onto a sizable smooth sheet of paper or oilcloth. It is important that the top of the hopper of the mill be fitted with a cover to prevent loss of seed during grinding. There should be practically no loss of material in grinding and if an appreciable amount of material is lost, the entire process should be repeated since the lost material is not necessarily representative of the whole.

Mix the ground sample of seed thoroughly using approved methods and equipment. When the ground sample is thoroughly mixed, the material shall be trans-

ferred to a well-stoppered bottle or container of proper size to hold the material tightly so as to prevent percolation or vertical segregation of the components.

Sec. 4: Second Moisture.

Determination. Weigh 5 grams of the fumed and ground sample into a shallow moisture dish and dry at 101°C. for 2 hours in an approved type oven. The dish when removed from the oven is covered, cooled in an efficient desiccator and weighed. Calculate loss in weight as percent moisture of the fumed and ground sample. (See appendix for approved equipment).

Sec. 5: Oil

(a) See appendix for approved equipment and reagents.

(b) Determination. Weigh accurately duplicate sample of 5 grams of the fumed and ground seed; wrap in a 150 mm. filter paper (S. & S. #597, or equivalent grade) and rewrap in a second paper or papers in such manner as to prevent escape of the meal, leaving the top of the second paper or papers open like a thimble. A small piece of absorbent cotton may be placed in the top of the thimble to distribute the dropping petrolic ether if desired. Place 25 ml. to 30 ml. of petrolic ether in a tared flask of 100 ml. capacity (smaller flask of not less than 50 ml. capacity may be used if preferred) and extract sample for 4 hours, the ether dropping in the center of the thimble at a rate of at least 150 drops per minute. The volume of the solvent should be kept approximately constant. At completion of the extraction period, the solvent is evaporated until no trace remains. The flask and contents shall be cooled to room

temperature and weighed. The last traces of the ether are sometimes difficult to detect by odor and, in case of doubt, evaporate for 30 minutes or longer until constant weight is obtained after cooling. Calculate the oil content as shown in the following example:

Example of Calculation

Petrolic ether extract	1.025 grams
Original moisture plus total foreign matter up to and including 1% 12.2% plus 0.8%	13.0%
Second moisture	2.6%
Total foreign matter up to and including 1%	0.8%
Weight of sample	5.00 grams

$$\text{Percent Oil} = \frac{1.025}{5} \times \frac{(87)^*}{(97.4)} = 18.3\%$$

* Factor to convert from dry basis to original moisture basis is calculated thus:

$$F = \frac{100 - (12.2 \text{ plus } 0.8)}{100 - 2.6} = 89.32$$

Sec. 6: Ammonia

(a) See appendix for approved equipment and reagents.

(b) Determination.

(1) Digestion Procedure. Digest 1.7034 or 1.401 grams of the fumed and ground sample in a Kjeldahl flask with approximately 0.5 gram of metallic mercury or 0.7 gram of mercuric oxide, 10 grams of sodium or potassium sulphate and 25 ml. of concentrated sulphuric acid. Place the flask in an inclined position and heat below the boiling point of the acid from 5 to 15 minutes, or until frothing has ceased. Increase the temperature and continue digestion until the liquid becomes colorless, or until complete digestion is obtained (generally 45 minutes to 1 hour is sufficient). The procedure is the same from this point on as in the regular Kjeldahl method, except that no potassium permanganate is added.

(2) Distillation Procedure. After cooling, add about 250 ml. to 300 ml. of water, a few granules of zinc to keep the contents from bumping, 25 ml. of a 4 percent solution of potassium or sodium sulphide, or a sufficient amount to precipitate all of the mercury. After mixing thoroughly, add 50 ml. to 60 ml. of caustic soda solution (specific gravity of 1.50), or sufficient amount to make strongly alkaline, pouring the solution down the side of the flask so that it does not mix at once with the acid solution. Connect the flask with a condenser of block tin or heat-resistant glass, mix the contents of the flask by shaking, and distill into an accurately measured quantity of standard sulphuric acid solution to which has been added 50 ml. of distilled water, until at least 200 ml. of the distillate is obtained, taking care that the delivery tube reaches below the level of the standard acid. (The strong caustic soda solution and the solution of sulphide may be added together if desired). Add about 1 ml. of a 0.2% solution of a proper indicator to the distillate (if methyl red is used, the solution may be titrated hot), and titrate with a standfixed alkali solution. Calculate results and report as ammonia.

(3) Blank Correction. Make blank test on all reagents and correct the titration of the above distillate accordingly.

(4) If the ammonia percentage found in the fumed and ground sample is less than 3.70% or more than 4.50%, a second determination shall be made and if those two determinations do not agree within 1/10 of 1%, two additional determinations shall be made and the average of the two or three determinations agreeing most closely shall be used in the calculations.

Example of Calculation

Quantity of 0.5N H_2SO_4 measured into flask	10.00 ml.
Quantity of 0.5N H_2SO_4 for blank test on reagents	0.06 ml.
Quantity of 0.25N NaOH used in titration	2.68 ml.

$$\frac{10.00 - 0.06}{2} - \frac{2.68}{4} = 4.30\% \text{ ammonia in fumed seed}$$

Original moisture	8.1%
Foreign matter up to and including 1%	0.9%
Moisture in fumed and ground seed	2.0%

$$\frac{4.30 \times 0.91}{0.98} = 3.99\% \text{ ammonia in original seed}$$

Sec. 7: Free Fatty Acids

Determination. Dry 200 grams of the original clean sample of seed for not less than 30 to 40 minutes at a temperature of 100°C. to 105°C. in an approved type oven and cool. Pass the cooled seed through an approved laboratory huller. Separate all of the meats from the hulls by screening. The meats shall be ground by passing the meats through an approved grinder and thoroughly mixed. Proper grinding and complete separation of the meats from the hulls are essential in order to obtain concordant results. Without undue loss of time, quarter the thoroughly mixed ground meats so as to obtain at least 40 grams of meats for extraction. Extract this portion by cold percolation as follows:

Place a disc from a Knorr Extraction Apparatus in a Butt tube and cover it with a layer of asbestos fibre suspended in petrolic ether, or pack the bottom of the tube with cotton. The cotton should extend approximately one-half inch into the small end of the tube and one-half inch above the bottom of the body of the tube, care being taken so as not to pack the cotton so tight as to prevent proper drainage. A satisfactory mat should not allow any of the meats to pass through, but should allow the extracting solvent to pass through at a rate of about 150

drops per minute. Place the sample of ground meats to be extracted in the prepared tube and add 50 ml. of petrolic ether, followed by two portions of 25 ml. each. Each portion of petrolic ether should be allowed to flow through the sample into the extraction flask before the following portion is added. Complete extraction of the oil is essential for accuracy of the determinations. Allow the extracted oil to remain on a steam bath for a sufficient period of time to completely remove all traces of the solvent from the oil. Weigh 7.05 grams of the oil after cooling into a titrating flask, add 30 ml. of neutralized approved alcohol, 1 ml. of 1% solution of phenolphthalein indicator (10 ml. of petrolic ether may be added if desired to sharpen the end-point) and titrate the free fatty acids of the oil with a standard alkali. The flask should be shaken vigorously during the titration and the end-point taken when a permanent pink color is obtained and which persists for at least one minute. Record the amount of standard alkali used in the titration and calculate the percent free fatty acids of the oil.

If the results indicate a free fatty acids content of 4.0% or higher, the complete test shall be duplicated.

Example of Calculation

$$\frac{28.2 \times \text{Normality of alkali} \times \text{ml. used}}{\text{Weight of oil used}} = \text{Percent Free Fatty Acids}$$

Sec. 8: Calculation of Analysis.

From the moisture determined on the whole seed as received, plus the total foreign matter up to and including 1.0%, and the moisture determined on the fumed and ground sample, the percentages of oil and ammonia are calculated back to the original moisture basis as received by the following formula:

M = Moisture in original seed.
FM = Total foreign matter up to and including 1.0%.
P = Moisture in the fumed and ground sample.
F = Factor to multiply by to reduce results from dry basis to the original basis as received.

$$\frac{100 - (M \text{ plus } FM)}{100 - P} = F$$

Example of Calculation

Percent of oil in fumed and ground seed	20.5
Percent of ammonia in fumed and ground seed	3.90
Percent of total foreign matter up and including 1.0%	0.8
Percent of moisture in original seed	12.2
Percent of moisture in fumed and ground seed	2.6
Factor to multiply by to reduce to original moisture basis	89.32

$$\frac{100 - (12.2 \text{ plus } 0.8)}{100 - 2.6} = 89.32$$

20.5 x 89.32 = 18.3% of oil
3.90 x 89.32 = 3.48% of ammonia

Sec. 9: General Instructions.

(a) All calculations shall be carried out to the third decimal place.

(b) Fractions of exactly one half shall be dropped when the next higher decimal figure is an even number and used to raise the next higher decimal figure if it is an odd number.

Example: 0.345 equals 0.34
0.335 equals 0.34

(c) Data on reports of seed analysis shall be expressed as follows:

Foreign Matter to	0.1%
Percent of Oil to	0.1%
Percent of ammonia to	0.01%
Free Fatty Acids, when 5.0% or under, to	0.1%
Free Fatty Acids, when over 5.0%, to	0.5%
Quantity Index to	0.01 units
Quality Index to	0.1 units

Grade to whole or half units, whichever the actual calculation is nearest.

(d) A sample certified by a licensed cottonseed sampler as hot or fermented shall not be designated as "Off Grade" unless the chemist shall find evidence of damage due to fermentation or heating.

(e) No certificate of the grade of a sample shall be issued until after the lapse of 20 hours after the receipt of the sample by the chemist.

(f) Each step in the analysis of samples of cottonseed shall be executed promptly and with a minimum of exposure to oxidation. Once begun, all analytical operations shall be continuous with no interruption or delay at any point.

(g) The form of cottonseed grade certificate shown on page 10 shall be used by all licensed cottonseed chemists. When linters have been included as a factor in determining the grade of cottonseed, the capital letter (L) in parentheses shall be placed immediately after the numerical grade designation on the cottonseed grade certificate. The certificates shall not contain advertising matter.

U. S.
(SEAL)
D.A.

UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Marketing Service

COTTONSEED GRADECERTIFICATE

A. B. C. LABORATORIES, cooperating
30 Wall Street, New York City

Issued at _____
Date _____

Submitted by _____
_____

Identified as _____

Point of Origin _____ County, _____ State

Sample certified by _____
Licensed Cottonseed Sampler No. _____

ANALYSIS		CALCULATIONS	
Total Foreign Matter (Reported by sampler & chemist)	_____ %	Quality Index Deductions: Acc't. Foreign Matter	_____ units
Moisture	_____ %	Acc't. Moisture	_____ units
Free Fatty Acids in Oil	_____ %	Acc't. Free Fatty Acids	_____ units
Oil	_____ %	Net QUALITY Index	_____
Ammonia	_____ %	QUANTITY INDEX	_____
Linters	_____ %	GRADE	_____

I hereby certify that the above analysis was made according to the laboratory methods approved by the Administrator, ~~Production and Marketing Administration~~ Agricultural Marketing SERVICE, and that the grade given is according to the Official Standards of the United States.

Certificate No. _____

_____
Licensed Cottonseed Chemist No. _____

Sec. 10: Cotton Linters - Option of Forced-draft Oven Method or Vacuum Method

1. Forced-draft oven method

(a) See appendix for approved equipment and reagents.

(b). Procedure. Dry duplicate portions of 50 grams of cottonseed, plus or minus 0.01 grams, for 30 minutes at 130°C., plus or minus 3°C. in an approved type oven. If the seed contain excessive moisture, they should be dried for 1 hour. Toward the end of this drying period absorb into the inner walls and bottom of a porous earthenware pot 2.0 ml. of concentrated hydrochloric acid (use 1.0 ml. for delinted cottonseed). The acid should be distributed all over the inside of the pot, and when absorbed the inside of the pot must appear dry, otherwise a new pot must be substituted. Place the dried seed in the pot, cover and place in an approved type of fuming oven which has been previously opened and ventilated for at least 5 to 10 minutes, and fume for 1 hour. The oven temperature should gradually rise to, but not exceed, 115°C. When properly fumed, the lint should be loose and brittle, but not scorched. Transfer the treated seed to an approved sieve or screen and carefully brush with a rotating or circular motion, using a round brush, by hand or approved machine, until all of the lint has been removed from the seed and passed through the screen. Transfer the delinted seed to metal boxes provided with close fitting covers, covers removed, and place in an approved oven and dry over-night at 101°C. Remove from the oven, cover, cool in a desiccator and weigh to the nearest 0.01 grams. Determine moisture in the original seed by the same method as specified in Sec. 2. Calculate and report in accordance with the following example:

Example of Calculation

A equals Weight of sample (50 grams)
B equals Weight of dry, delinted seed
C equals Moisture in original cottonseed
Residual Lint, 8% moisture basis (when 50 gram sample is used)

$$\% = \frac{2\,(A - B) - C}{0.92}$$

2. Vacuum method

(a) See appendix for approved equipment.

(b) Procedure: Weigh duplicate portions of 50 grams ±.01. Absorb 2 ml. of concentrated HCL in the bottom of vacuum type earthenware porous pots. (The acid should be distributed over the bottom of the pot. If the botton of the pot does not appear to be dry, another pot must be substituted). Place the weighed seed in the pot, cover and place in an approved type vacuum oven. Close oven door and allow vacuum to increase slowly until 14 ± 1" Hg vacuum is reached within 13 minutes. The temperature of the bottom of the oven will be thermostatically maintained at 158° C ± 2°. After 13 minutes in the oven, the vacuum will be increased so that a vacuum of 27 ± 1" Hg will be reached at the end of 12 minutes continued treatment. (The low vacuum tends to pull the HCL fumes out of the pot into the cottonseed; the higher vacuum pulls the fumes out of the cottonseed and out of the oven.) When a temperature of 160°C is maintained on the floor of the oven, the temperature within the pots will rise until approximately 90° is reached at the end of the fuming treatment.

With excess moisture cottonseed (more than 20 percent), the method is modified. The cottonseed is predried under vacuum for 10 minutes in vessels without covers. The HCL is absorbed into the inside of the vessel covers which are placed on the vessels containing the cottonseed. The samples are then fumed as outlined above.

Weigh the cottonseed to the nearest .01 gram. Transfer the seed to an approved delinting device for 1½ minutes or until all lint has been removed and passed through the screen. Weigh the delinted seed to the nearest .01 gram. Two-vacuum ovens and one brush machine have a capacity of 108 samples (in duplicate) in 8 hours or 324 samples (in duplicate) in a 24 hour period.

Calculate and report residual linters in accordance with the following example:

B = Weight of cottonseed after fuming treatment

C = Weight of delinted cottonseed

Residual linters, 8% moisture basis, in percent

$$= \frac{2(B-C)}{0.92}$$

Note: When the pots do not absorb the acid, they may be replaced with new pots or re-activated as follows:

Boil for approximately one hour in 10 percent NaOH solution. Rinse in water. Heat in a 1 percent solution of HCL for 20 minutes. Rinse in water. Dry overnight at 130°C. This treatment renders the pots as good as new and in some cases the absorption is actually increased.

Sec. 11: Cellulose Yield for cottonseed linters and hull fiber.

(a) See Appendix for approved equipment and reagents.

(b) Procedure. Place up to 5 lbs. of linters or hull fiber in an approved mixer, close the door, and rotate the mixer 3 to 5 minutes, depending on the length of the fiber. Three minutes is sufficient for second cut or hull fiber; 5 minutes for mill-run. After mixing, remove portions for moisture and cellulose yield determinations. Should the conditions of humidity and temperature in the laboratory be such as to cause changes in temperature during the mixing of the lint, the moisture determination should be made as soon as the sample is received in the laboratory. A 25 gram sample is weighed into a tared approved moisture dish and dried in an approved oven at 105°C. to 110°C. for 4 hours. Remove from the oven, cover and cool in a desiccator. Weight the cooled sample, calculate and report as moisture of the lint in accordance with Sec. 2., for moisture percentage. Weigh accurately 35 grams of the mixed sample into an approved digestion vessel. Add 525 ml. of standard 1.0% alkali solution, pressing the sample of lint into the alkali solution as the alkali is added (it is essential that all of the sample be wet). Stir the sample thoroughly, fasten the lid securely to the vessel and place in approved autoclave (pressure cooker). Fasten the lid of the autoclave securely and increase the steam pressure up to 105 lbs. (it is advisable to blow steam through the autoclave before fastening the lid to close in order to be sure that all air has been removed). Observe the temperature closely throughout the digestion period, and digest for 3 hours after the temperature has reached 341°F. and then reduce the steam pressure gradually. Be careful not to allow the steam pressure to drop during the digestion period. Remove the sample from autoclave, add a small amount of water and pour the mixture directly into the lower half of an approved lint washer. Rinse the sample container once in order to assure the complete

removal of all fiber into the washer. Attach the upper portion of the cylinder securely and start the washer, opening the water valve when the screened portion of the washer reaches the bottom of its rotation. Observe the time at which the water is turned on, maintain the water pressure constant at 22 lbs. per square inch, and at a rate of 3.9 to 4.0 gallons per minute. Wash the sample for 5 minutes. Close the water valve and stop the washer when the screened end of the washer reaches the bottom of its rotation, keeping the cylinder slightly off the vertical position to insure complete drainage. When the flow of water from the cylinder has stopped, reopen the water valve for a few seconds in order to wash any adhering fibers from the walls of the cylinder. Remove the lower half of the washer containing the sample, carefully remove the sample from the screen and compress it to remove as much water as possible. Place the sample in an approved moisture dish, dry in an approved oven at 105°C. to 110°C. for 16 hours, preferably over-night. Remove from the oven, cover, cool to room temperature and weigh. Calculate the report percentage of dry cellulose or any desired moisture basis in accordance with the following example:

Example of Calculation

$$\% \text{ Moisture} = \frac{\text{Loss in Weight} \times 100}{\text{Weight of Sample}}$$

$$\% \text{ Cellulose yield (dry cellulose) received basis} = \frac{\text{Weight of Dry Residue} \times 100}{\text{Weight of Sample}}$$

$$\% \text{ Cellulose yield (desired moisture basis)} = \frac{A\ (100 - \% \text{ moisture desired})}{100 - \% \text{ moisture in sample analyzed}}$$

A = % dry cellulose yield determined above.

APPENDIX "A"

APPROVED EQUIPMENT AND REAGENTS: -

Sec. 1 (a): Friction-top cans of 155 cubic inch capacity.
or
Paper bags 7-1/2" x 3" x 14-1/2", 1/90 Asphalt Laminated or 1/60 Duraloid, sewn, open mouthed, bottoms dipped in wax.

Sec. 1 (c): 6-mesh Screen.

Torsion Balance, capacity 2,000 grams.

*USDA Type Laboratory Cottonseed Cleaner-Mixer (Mfg. by Custom Scientific Instruments, Inc., Arlington, New Jersey
or
MacLellan Mixer No. 00-S.
or
*Henry Mixer (mfg. by Davidson-Kennedy Company, Atlanta, Ga.).

Sample containers for use in making analysis.

Air-Tight bottles or containers, wide-mouth, for preserving portion of sample for moisture determinations.

Sec. 2: Shallow Moisture Dishes with Covers, Official A.O.C.S. or equivalent.

Analytical Balance.

Forced-draft Circulatory Oven approved by A.O.C.S. (Despatch Oven and Freas (Precision-Freas) Oven only manufactured type ovens approved; converted DeKotinsky Ovens also approved).

Desiccator, minimum 10" diameter or larger.

Sec. 3 (a): Forced-draft Circulatory Oven (same as specified in Sec. 2).

Fuming Oven: double-walled tank constructed of sheet iron or copper, seams welded or brazed, open at top, removable cover, inner compartment approximately 7-1/4" deep and 7" wide, equipped with removable tray large enough to hold two rows of fuming pots, tray to be so constructed so as not to rest directly on bottom of the compartment, length of compartment recommended approximately 18";

space between walls and bottom of tank should be filled with a mineral oil which can be heated to about 175°C., space to be fitted with breather pipe and drain; cover to have two or three holes 3/4" diameter for ventilation; inner compartment and space between walls of tank should be fitted with thermometers in order to control temperatures of oil and fuming compartment; heating of oven to be by gas or electricity.

or

*Henry Forced-draft Fuming Oven equipped with thermostatic control, heat being furnished by gas jets (mfg. by Davidson-Kennedy Co., Atlanta, Ga.).

or

*USDA type vacuum fuming oven (mfg. by Lab-Quip Mfg. Co., Leland, Miss.)

Fuming Pots: unglazed porous earthenware pots with (1) outside dimensions 3 1/8" diameter, 3 3/8" high, inside dimensions 2 5/8" diameter, 3 1/8" depth, or (2) new type pot with inside dimensions 5" diameter and 1" depth (mfg. by Niloak Pottery Company, Little Rock, Ark.), pots to be fitted with covers.

Grinding Mills: Bauer Bros. No. 148 Laboratory Mill, using Precision-ground No. 6912 plates, speed of 3600 r.p.m., hopper fitted with cover (mfg. by the Bauer Bros. Company, Springfield, Ohio).

*Mixer; Henry Velocity Mixer for ground sample (mfg. by A. S. Aloe Company, St. Louis, Mo.).

or

a 1/2 gallon Mason Fruit Jar with screw type cover and large rubber stopper.

Air-tight bottles or containers, wide mouthed, screw caps for ground samples.

Concentrated Hydrochloric Acid.

Sec. 4: Shallow Moisture Dishes with Covers (same as specified in Sec. 2).

Forced-draft Circulatory Oven (same as specified in Sec. 2).

Analytical Balance.

Desiccator (same as specified in Sec. 2).

Sec. 5 (a): Butt Extraction Apparatus using Allihn Condensers with 12" jackets, fitted with tapered cork connections.

Soxlet Extraction Flasks, 50 ml. or 100 ml, capacity as desired.

Filter Paper, 150 mm., S. & S. No. 597, Reeve Angel No. 211, Whatman No. 2 or equivalent.

Absorbent Cotton, Free of petrolic ether extract.

Analytical Balance.

Hot Water (steam) Bath or Electric Hot Plate covered with 1/8" asbestos sheet.

Petrolic Ether of the following specifications:

Initial boiling temperature	Not less than 35°C.
Initial boiling temperature	Not more than 38°C.
Dry flask end point	Not less than 52°C.
Dry flask end point	Not more than 60°C.
At least 95% distilling under 54°C.	
Not more than 60% distilling under 40°C.	
Specific Gravity at 60°F.	0.630 to 0.660
Color	Water white.
Evaporation residue, 100 ml.....	Not more than 0.0011 grams
Doctor Test	Sweet.
Copper Strip Corrosion test	Non-corrosive.
Unsaturated compounds	Trace only permitted.
Residue in distilling flask	Neutral to methyl orange.
Blotter Strip Odor test.....	Odorless within 12 minutes.
Aromatic compounds	No nitro-benzene odor.
Saponification value	Less than 1.0 mg. KOH per 100 ml.

Distillation test performed as directed in A.S.T.M. Designation 216-32.

Evaporation Residue: add 0.25 grams of stearin or similar hard fat, previously dried to constant weight at 101°C., to 250 ml. of

petrolic ether, evaporate the ether on a steam bath and dry the residue to constant weight at 101°C.. The increase in weight shall not exceed 0.003 grams.

Copper Strip Corrosion test: insert a small strip of polished copper in a distillation flask containing petrolic ether. There should be no appreciable darkening of the copper during distillation.

Unsaturated compounds: determine as directed on page 154 of the Analytical Edition of I. & E. C., March, 1938. Trace only permitted.

Blotter Strip Odor test: immerse a strip of white unglazed blotting paper (1" x 4" x 0.166") in petrolic ether for 30 minutes; dry at room temperature in still air for 12 minutes. The paper should remain odorless.

Aromatic compounds: add 5 drops of petrolic ether to 40 drops of H_2SO_4 (sp.gr. 1.84) and 10 drops of HNO_3 (sp.gr. 142) in a test tube; warm for 10 minutes and then cool for 30 minutes; transfer to a shallow dish, dilute with distilled water. There should be no nitrobenzene odor.

Sec. 6 (a): Kjeldahl Digestion and Distillation Apparatus, complete with heat source (gas or electricity), traps, and block tin or equivalent non-corrosive tubing condensers.

Kjeldahl Digestion Flasks, 800 ml, or 650 ml. capacity as desired.

Distillate receiving Flasks, 500 ml. capacity or any convenient size.

Metallic Mercury or Mercuric Oxide, A.C.S. grade.

Zinc metal, granular, 20-mesh.

Potassium or Sodium Sulphate, A.C.S. grade.

Sulphuric Acid, specific gravity 1.84, reagent grade.

Sodium Hydroxide Solution, specific gravity 1.50, technical grade.

Sodium Hydroxide Solution, 0.25N, accurately standardized. (see Note)

Sulphuric Acid Solution, 0.5N, accurately standardized. (See Note)

Distilled Water.

Methyl Red Indicator Solution, 0.2% in alcohol (SDA 30 or Ethyl Alcohol)

Potassium or Sodium Sulphide Solution, 4.0% in water.

NOTE: Other normalities of accurately standardized Sodium Hydroxide Solution and Sulphuric Acid Solution may be used if desired by analyst.

Sec. 7: Laboratory Huller or Bauer Bros. Mill No. 148 (as specified in Sec. 3) with the plates adjusted to just break the seed.

Perforated porcelain or metal disc, 17 mm. diameter, with 1 mm. holes spaced 1 mm. apart.

Butt Tubes.

Asbestos Fiber suspended in petrolic ether.

Absorbent Cotton.

4- to 6-mesh Screen.
or
*Henry Hull and Meats Separator (mf'g. by Davidson-Kennedy Co., Atlanta, Ga.)

Barrow-Agee Meats Grinder, equipped with Russwin No. 1 or Universal No. 71 food chopper with 16-tooth blade or Universal No. 1 food chopper with 12-tooth blade.

Oil-sample Bottles.

Extraction Flasks of 150 ml. capacity.

Petrolic ether (specifications same as in Sec. 5 (a)).

SDA Formula 30 alcohol (or SDA 3A), Isopropyl alcohol or Ethyl Alcohol (whichever alcohol is used, the alcohol must be neutralized with NaOH solution until indicator shows faint pink color before adding sample of oil).

Phenolphthalein Indicator Solution, 1.0% in 95% Ethyl Alcohol or SDA Formula 30 Alcohol.

Sodium Hydroxide Solution, 0.25N, accurately standardized.

Analytical Balance.

Torsion Balance.

Sec. 10 (a): Forced-draft Circulatory Oven (same as specified in Sec. 2).

Fuming Oven (same as specified in Sec. 3 (a)).

Fuming Pots (same as specified in Sec. 3 (a)).

Shallow Moisture Dishes (same as specified in Sec. 2) and Metal Moisture Dishes, 3-3/4" x 1", with tight fitting slip-cover covers.

Sieve, Tyler 35-mesh (U. S. No. 40).

Round Brush (a window washing brush is satisfactory).

Note: specification for a suitable delinting machine may be obtained from the U. S. Department of Agriculture, PMA., Washington, D. C. (Cotton Branch). Other satisfactory machines are as follows: Rottger-Atkinson Brushing Machine (c/o T. L. Rottger, Buckeye Cotton Oil Co., Memphis, Tenn.) and Fort Worth Fumed Lint Removal Machine (c/o C.L. Manning, Fort Worth Laboratories, Fort Worth, Texas)

Desiccator (same as specified in Sec. 2). Note: Calcium Chloride is not considered a suitable desiccant: see A.O.C.S. approved desiccants.

Sec. 11 (a): Forced-draft Circulatory Oven (same as specified in Sec. 2).

Mechanical Lint Mixer as approved by A.O.C.S.

Mechanical Washer (mfg. by William E. Ellis & Sons, Memphis, Tenn.) to A.O.C.S. specifications.

Pressure Cooker or Autoclave which will maintain an internal steam

pressure of 105 lbs. plus or minus 1 lb. per square inch (equivalent to 341°F. or 171.6°C.).

Digestion Vessels, iron containers (glue pots) capacity 1000 ml. (A.O.C.S. specifications).

Moisture Dishes, aluminum, capacity 30 cubic inches, with tight fitting covers.

Metal Moisture Dishes (same as specified in Sec. 10 (a)).

Desiccator (same as specified in Sec. 2 using a desiccant as specified in Sec. 10 (a)).

Sodium Hydroxide Solution, 1.0% NaOH by weight, accurately standardized.

For description of items marked with asterisks (*) see Appendix "B"

APPENDIX "B"

Description of Newly Approved Equipment:

Sec 1 (e): Henry Mixer for whole cottonseed sample as received.

This mixer consists of a hopper of approximately 5 gallon size, so designed that a set of four paddles will scoop the middle third of the sample from the bottom, permitting the other two-thirds to fall to the bottom As paddles lift the middle third of the sample, it is re-distributed by falling over the other two-thirds of the sample which have slipped to the bottom because of the slant given the sides of the hopper. Approximate time of operation to give a thorough mixing of the sample is 1 minute.

USDA Type Laboratory Cottonseed Cleaner-Mixer: This device is a paddle type cleaner-mixer which provides a means for mechanically agitating the cottonseed in a circular metal container having a flat bottom with slotted perforations. The cleaning principle employed is similar to that of the hand cleaning method now used wherein the cottonseed samples are rubbed back and forth across a screen. Power is supplied by a small geared motor. The paddle assembly consists of a round disc with rubber paddles mounted on its lower side. A hand wheel is provided for raising and lowering the cleaning paddle assembly so that it is out of the way when elevated for ease in removing cottonseed. A conveniently located lever operates a lock which holds the paddle assembly in either an up or down position. The hand wheel shaft is spring loaded to assist the operator in raising the paddle assembly.

Sec. 3 (a): Henry Forced-draft Fuming Oven equipped with thermostatic control for fuming of cottonseed for oil and ammonia determinations. This oven is essentially a forced-draft, circulatory, gas-fired, constant temperature oven, so designed that the circulated air is preheated and delivered to the fuming chamber at a constant predetermined temperature. This permits the temperature of the fuming chamber to drop when the cold pots are introduced into the oven and to gradually rise to the maximum temperature as the fuming progresses. The fuming chamber is designed to accommodate four trays carrying 12 approved fuming pots each.

USDA Type Vacuum Fuming Oven: The vacuum fuming equipment consists of an insulated box type vacuum oven electrically heated, thermostatically controlled, 1800 watts, and a self-supplying centrifugal water pump to provide vacuum with water aspirators. By use of a bleeder valve, a vacuum of 17 inches mercury is provided in the oven. Highest vacuum obtained is about 27 inches of mercury. Each individual oven has a capacity of six samples. Additional oven units may be added for a greater capacity.

Sec. 3 (a): Henry Velocity Mixer for mixing of ground cottonseed samples for oil and ammonia determinations.

This mixer consists of an open sample cup revolving on a vertical axis at speeds of approximately 100 to 500 r.p.m., maximum speed recommended for cottonseed, and a baffle blade mounted rigidly at one side of the cup for directing the flow of the sample. In operation, the sample in the rapidly revolving cup assumes the properties of a fluid in flow. As the velocity is reached, the baffle blade at the side of the cup re-directs this stream to produce essentially perfect mixing. The mixer is built with a speed reducer motor of 1/8 h.p. capacity enclosed in a suitable housing. Speed is controlled by a knob and dial graduated 0 to 100 units in 2 unit sub-divisions. The sample cup is made of stainless steel and is mounted directly on the motor spindle through an opening in the top of the motor housing. The cup is removable so as to easily facilitate the removal of the mixed sample. Approximate time of operation to give a thorough mixing of the sample is 1 minute with the cup revolving at full speed.

Sec. 7: Henry Hull and Meats Separator for separating meats from hulled seed for use in F.F.A. determination.

This separator consists of a square hopper equipped with rotary beater turning so as to beat the sample against a curved screen (4 to 6 mesh) until the meats have been separated and screened into a chute which passes them back to the sample container. The hulls are discharged from the hopper by revolving the whole mechanism on the beater shaft and dumping into any properly placed receiver or container. Complete separation is obtained in approximately one minute.

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